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(54) Title: REMOVAL OF POST-ETCH RESIDUES IN SEMICONDUCTOR PROCESSING

(57) Abstract: A method of cleaning etching residue from a substrate comprising a low k dielectric material comprising the steps of : contacting the substrate with a composition comprising H₂SiF₆ or HBF₄, organic solvent, an amine, a corrosion inhibitor and water, said composition being capable of exfoliating post-etch residues and of minimizing oxide loss, and wherein said composition has a pH below 7.

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processes produce undesirable residues from the interaction of the plasma gases, reacted species and the photoresist. The composition of the residue is influenced by the type of etching, the resist, the underlying substrates, and the process conditions utilized.

[0005] If etching residue is not removed from the substrate, the residue can interfere with subsequent processes involving the substrate. The effect of poor cleaning results in low device yield, low device reliability, and low device performance.

[0006] To fulfill the demand for faster processing speed from semiconductor, the conventional Al or Al alloy used as the interconnection material has been replaced with Cu or Cu alloy, typically using a known damascene process. A barrier film, which may be silicon nitride, and a Low-k film are successively formed on the substrate, and a resist mask is then formed. The common low-K dielectrics include CORAL, tetraethylorthosilicate (TEOS), SiOC, porous MSQ, SiON, and boron phosphosilicate glass (BPSG). New low-k films having a lower dielectric constant than the p-TEOS film include a film formed of inorganic material such as porous silica or the like, a film formed organic material such as polyimide, polyarylene or the like, and a film formed of a mixture of the above-mentioned inorganic and organic materials. Next, the exposed low-k film is dry etched to expose the barrier film, so that a via hole is formed. At this time, reactive products of the gas used for the dry etching and the Low-k film and the resist film accumulate in the via hole as resist residue. Then, the resist film is removed by plasma ashing, leaving a modified film on the surface of Low-k film according to the reaction of the resist to heat and plasma during ashing. Then the resist residue is removed by processing with a fluoride-based cleaning composition. To ensure the removal of the resist residue, usually cleaning compositions have hydrofluoric acid that aggressively attacks the via sidewall of the dielectric and therefore changes the dimensions of the device, as taught by Ireland, P., *Thin Solid Films*, 304, pp. 1-12 (1997), and possibly the dielectric constant likely to evenly etch the insulating film has been used, and the via holes are enlarged. D. It is not uncommon for the HF to also attack the dielectric material. Such attack is not desirable (see Lee, C. and Lee, S., *Solid State Electronics*, 4, pp. 92 1-923 (1997)). Subsequently, a resist film patterned for trench formation is formed on the Low-k film, and, using the resist mask, the Low-k film is dry etched down to its intermediate position to form a trench. Resist residue that is the reactive product of the gas used for the dry etching and the Low-k film accumulates in the via hole and trench. The resist film is removed by plasma ashing, and resist residue is removed by processing with a conventional fluorine type

the etch rate is only 0.12 Å/min, as reported at NSF/SRC Eng. Res. Center, Environmentally Benign Semiconductor Manufacturing, Aug. 5-7, 1998, Stanford University. Several low-k materials can be altered by water. Polar organic solvents, typically amides, ethers, or mixtures thereof, can partially or completely reduce water. These solvents may be necessary for some substrates, but they are expensive and pose disposal problems.

[0011] Dilute compositions can lose effectiveness after repeated use, for example in baths. Recent information also indicates that the dilute HF solutions can be ineffective for cleaning the newer CF_x etch residues, as taught by K. Ueno et al., "Cleaning of CHF_3 Plasma-Etched $\text{SiO}_2/\text{SiN}/\text{Cu}$ Via Structures with Dilute Hydrofluoric Acid Solutions," *J. Electrochem. Soc.*, vol. 144, (7) 1997. Contact holes opened on to the TiSi_2 have also been difficult to clean with dilute HF solutions since there appears to be an attack of the underlying TiSi_2 layer. There may also be difficulty with mass transport of the chemicals in the narrow hydrophilic contact holes, as taught by Baklanov, M.R. et al., *Proc. Electrochem. Soc.*, 1998, 97-35, pp. 602-609.

[0012] Accordingly, there exists a need to develop improved cleaning compositions to efficiently clean a variety of deposits from a wide variety of substrates. Particularly in the field of integrated circuit fabrication, it should be recognized that the demands for improved cleaning performance with avoidance of attack on the substrates being cleaned are constantly increasing. This means that compositions that were suitable for cleaning less sophisticated integrated circuit substrates may not be able to produce satisfactory results with substrates containing more advanced integrated circuits in the process of fabrication. The cleaning compositions should also be economical, environmental friendly and easy to use.

[0013] In a first embodiment, the present invention relates to compositions capable of exfoliating post-etch residues and of minimizing oxide loss and including: (a) H_2SiF_6 , HBF_4 , or both; (b) an organic solvent; (c) optionally an amine; (d) a corrosion inhibitor; and (e) water, or in another embodiment a composition including: H_2SiF_6 , water, and optionally a corrosion inhibitor. In preferred embodiments, the composition has H_2SiF_6 , and the pH lower than 7.

[0014] In one embodiment, the composition contains: (a) H_2SiF_6 , HBF_4 , or both, at a total concentration from about 0.001 % to about 5 %, e.g., from about 0.3% to about 4%, by weight of the composition; (b) the organic solvent at a concentration from about 50 % to about 98 % by weight of the composition; (c) the amine at a concentration less than about 1.5

[0017] Unless otherwise specified, all percentages expressed herein should be understood to refer to percentages by weight. Also, the term "about," when used in reference to a range of values, should be understood to refer to either value in the range, or to both values in the range.

[0018] The present invention is directed to a composition effective in post-etch resist residue removal comprising: (a) H_2SiF_6 or HBF_4 ; (b) an organic solvent; (c) optionally an amine; (d) a corrosion inhibitor; and (e) water. The pH of the composition is lower than 7.

[0019] In the composition of the present invention, a conventional fluoride compound typically included in the cleaning solution can advantageously be replaced with a fluoride compound comprising H_2SiF_6 and HBF_4 . If the amount of H_2SiF_6 or HBF_4 contained in the composition is too low, the resist residue exfoliation power of the composition is too weak. Accordingly, in one preferred embodiment, the composition of the present invention contains H_2SiF_6 or HBF_4 at a concentration from about 0.001% to about 5% by weight, preferably from about 0.005% to about 3% by weight, for example from about 0.01% to about 2.5% by weight, or from about 0.1% to about 2% by weight of the composition. In a more preferred embodiment, the composition of the present invention contains H_2SiF_6 at a concentration from about 0.3% to about 4% by weight, preferably from about 0.5% to about 2% by weight, for example from about 0.7% to about 1.7% by weight of the composition.

[0020] In one embodiment, the composition contains: from about 0.001 % to about 5 %, e.g., from, by weight of H_2SiF_6 ; optionally a corrosion inhibitor at a concentration from about 0.001 % to about 10 % by weight of the composition; and water. The amount of water is preferably at least about 90%, e.g., at least 98%, by weight. In another embodiment, the composition further contains from about about 0.05 % to about 7 % by weight of the corrosion inhibitor.

[0021] In addition, many embodiments of the composition of the present invention can also contain an organic solvent. Examples of such organic solvents include, but are not limited to: amides such as N,N-dimethylformamide, N,N-dimethylacetamide, N,N-diethylformamide, N,N-diethylacetamide, N-methylformamide, and N-methylacetamide; pyrrolidones such as N-methyl-2-pyrrolidone, N-ethyl-2-pyrrolidone, and N-hydroxyethyl-2-pyrrolidone; imidazolidinones such as 1,3-dimethyl-2-imidazolidinone, and 1-3-diethyl-2-imidazolidinone; alkyl ureas such as tetramethyl urea, and tetraethyl urea; polyhydric alcohols and their derivatives such as ethylene glycol, ethylene glycol mono-methyl ether, ethylene glycol mono-ethyl ether, ethylene glycol mono-butyl ether, ethylene glycol mono-methyl ether acetate, ethylene glycol mono-ethyl ether acetate, diethylene glycol, diethylene glycol mono-

aminobenzene), hydroxylamine, hydroxylamine derivatives; and the like; and combinations thereof.

[0024] A preferred alkanolamine is a two carbon atom linkage alkanolamine. The generic two carbon atom linkage alkanolamine compounds suitable for inclusion in the invention have the structural formula $N(X, Y)-C(R_1, R_1')-C(R_2, R_2')-Z-O-R_3$ wherein R_1, R_1', R_2, R_2' , and R_3 are, independently in each case, hydrogen or a linear, branched, or cyclic hydrocarbon containing from 1 to 7 carbon atoms; wherein Z is a group having the formula $-(Q-CR_1R_1'-CR_2R_2')_m-$, such that m is a whole number from 0 to 3 (i.e., when $m=0$, there is no atom between the $-CR_2R_2'$ group and the $-OR_3$ group in the formula above), R_1, R_1', R_2 , and R_2' may be independently defined in each repeat unit, if $m>1$, within the parameters set forth for these moieties above, and Q may be independently defined in each repeat unit, if $m>1$, each Q being independently either $-O-$ or $-NR_3-$; and wherein X and Y are, independently in each case, hydrogen, a C1-C7 linear, branched, or cyclic hydrocarbon, or a group having the formula $-CR_1R_1'-CR_2R_2'-Z-F$, with F being either $-OR_3$ or $-NR_3R_4$, where R_4 is defined similarly to R_1, R_1', R_2, R_2' , and R_3 above, and with Z, R_1, R_1', R_2, R_2' , and R_3 defined as above, or wherein X and Y are linked together form a nitrogen-containing heterocyclic C4-C7 ring.

[0025] In a preferred embodiment, the composition contains a two-carbon atom linkage alkanolamine compound, in which m is greater than or equal to 1. In another preferred embodiment, the composition contains a two-carbon atom linkage alkanolamine compound, in which m is 1 and R_1, R_1', R_2, R_2' , and R_3 are all hydrogen or a C1-C4 linear or branched hydrocarbon. In a more preferred embodiment, the composition contains a two-carbon atom linkage alkanolamine compound, in which: m is 1; R_1, R_1', R_2, R_2' , and R_3 are all hydrogen or a C1-C4 linear or branched hydrocarbon; and Q is $-NR_3$. In a most preferred embodiment, the composition contains a two-carbon atom linkage alkanolamine compound, in which: m is 1; R_1, R_1', R_2, R_2' , and R_3 are all hydrogen; X and Y are, independently, hydrogen or a linear or branched C1-C4 hydrocarbon; and Q is $-NH-$, $-NCH_3-$, or $-N[(C_2-C_4) \text{ linear or branched hydrocarbon}]$.

[0026] Hydroxylamines and hydroxylamine derivatives according to the invention can be characterized as having the following formula $N(X, Y)-O-R_3$ wherein R_3 is hydrogen or a linear, branched, or cyclic hydrocarbon containing from 1 to 7 carbon atoms; and wherein X and Y are, independently, hydrogen or a linear, branched, or cyclic hydrocarbon containing from 1 to 7 carbon atoms, or wherein X and Y are linked together form a nitrogen-containing

limited to: nitrate salts of ammonium; hydrocarbon-substituted ammonium nitrate salts; benzotriazole; 2,4-pentandione dioxime; 1,6-dioxaspiro[4,4] nonane 2,7-dione (di-ether); thiourea; ammonium bisulfite; choline salts, e.g., bisulfite, nitrate, hydroxide, or the like, or a combination thereof; bischoline salts, e.g., bisulfite, nitrate, hydroxide, or the like, or a combination thereof; trischoline salts, e.g., bisulfite, nitrate, hydroxide, or the like, or a combination thereof; glycerol; sorbitol; gelatine; starch; phosphoric acid; silicic acid; polyethylene oxide; polyethylene imine; benzotriazole; gallic acid or gallic acid esters; glycolic acid or glycolic acid esters; sugar alcohols such as traitol, erythritol, adonitol, xylitol, teritol, idetol, and dulcitol; and the like; and combinations thereof.

[0031] In one preferred embodiment, the composition contains the corrosion inhibitor at a concentration from about 0.001% to about 10% by weight, preferably from about 0.05% to about 7% by weight, for example from about 0.1% to about 5% by weight, from about 0.05% to about 2% by weight, from about 4% to about 7% by weight, or from about 4.5% to about 5% by weight of the composition.

[0032] The pH of the present composition is lower than 8.5 and is typically lower than 7. In a preferred composition, the pH can be from about 1 to about 6, more preferably from about 1.9 to about 5.

[0033] When necessary or as desired, the pH can be adjusted by using pH adjusting agents including, but not limited to: inorganic acids such as sulfuric acid, nitric acid, hydrochloric acid, and phosphoric acid; organic acids such as acetic acid, propionic acid, oxalic acid, glycolic acid, and citric acid; ammonia or ammonium salts such as hydroxide; organic amines such as ethylenediamine and their respective ammonium salts; quaternary (e.g., C1-C10 hydrocarbon-substituted) ammonium bases; and the like; and combinations thereof (including pH buffers). The amount of said pH adjusting agent added can be readily determined by the persons of ordinary skill in the art according to the specific components of the composition. A preferred acidic pH adjusting agent is phosphoric acid.

[0034] A manufacturing method of a semiconductor device according to the present invention includes the step of forming a metal film having copper as its main component on a semiconductor substrate, the step of forming an insulating film such as a Low-k film thereon, the step of forming a resist film further thereon, the step of providing a hole or a trench in the insulating film by way of dry etching using the resist film as a mask, the step of removing the resist by gas plasma processing or heat treatment, and the step of removing, using the cleaning composition for removing resists as described above, resist residue generated due to reaction between etching gas and the resist film and the insulating film at the time of dry

[0038] In one embodiment, the substrate can be immersed in the composition according to the invention. In some cases, when a production line progress is temporarily interrupted, those substrates exposed to the residue removers of the present invention with the resultant very low etch rates can be salvaged, while those substrates immersed in more aggressive formulations are destroyed. In an alternate embodiment, the composition according to the invention can be applied to a surface of the substrate, *e.g.*, by spraying on, applying droplets, coating, overcoating (with the excess composition running off the substrate), or the like, or a combination thereof.

[0039] There is no specific limit as to the time and temperature at which the composition of the present invention may be used in practice. For example, both the exfoliation time and exfoliation temperature can be readily determined by the persons of ordinary skill in the art based upon factors such as the condition of resist residues and specific components to be used in the composition.

[0040] This invention is also directed to a method of exfoliating the post-etch resist residues, comprising: providing a substrate with resist residues resulting from etching; contacting the substrate with the composition of the present invention for a time and at a temperature sufficient to cause the composition to substantially remove the resist residues; and rinsing the substrate.

[0041] The rinsing step can be carried out by using water soluble organic solvents, *e.g.* isopropanol, mixture of water soluble organic solvent and water, or using water only (*e.g.*, extra pure, distilled-deionized, or double deionized water).

[0042] EXAMPLES: Certain embodiments of this invention, as well as certain advantages of this invention, are illustrated by the following non-limiting examples. Although limited number of examples are disclosed herein, it will be apparent to those skilled in the art that many modifications, both to materials and methods, may be practiced without departing from the purpose and interest of this invention.

[0043] Example 1: A composition A was prepared according to the ingredients in Table 1. The pH of the solution was about 1.9. TEOS (tetraethoxysilane) and AlCu substrates were etched with a chlorinated plasma in a commercially available etcher. The resulting metal wafers with residues were cut into small sample pieces, and then the sample pieces were immersed into the solution of Table 1 and were cleaned for about 5 minutes at about room temperature. The sample pieces were taken out, rinsed with deionized water and dried with

[0044] Example 2: A composition B was prepared according to the ingredients in Table 3. The starting pH of the solution was 4.2. TEOS and AlCu substrates were etched with a chlorinated plasma in a commercially available etcher. The resulting metal wafers with residues were cut into small sample pieces, and then the sample pieces were immersed into the solution of Table 1 for cleaning for 24 hours at room temperature. The sample pieces were taken out, rinsed with deionized water and dried with the flow of N₂. SEM was performed with a Hitachi 4500 FE-SEM for evaluating cleaning and oxide loss. An excellent resist removal was observed. The observed oxide loss is summarized in Table 4.

Table 3. Composition B

Component	Amount Used (grams)	Percent by Weight
H ₂ SiF ₆	0.2	0.25
NMP	75.5	76
MEA	0.1	0.1
Xylitol	4.7	4.7
H ₂ O	18.7	Balance

Table 4. Observed Oxide Loss

Sample	Thickness Before Treatment (Å)	Thickness After Treatment (Å)	Change (Å)
TEOS #1	3019	3016	3
TEOS #2	3010	3006	4
TEOS #3	2989	2988	1
AlCu #1	5119	5024	95
AlCu #2	5189	5085	104
AlCu #3	5145	5009	136

[0045] The TEOS (oxide) loss was very low, and metal loss was low, for the 24 hour period.

[0047] The compositions in Table 5 were useful in cleaning low-K material TEOS in a time of between 30 seconds and 1 minute. These compositions are preferred because the lack of solvents makes them environmentally friendly and low-cost, and there is no bath life stability issues. However, the amount of H_2SiF_6 is beneficially below 0.5%, for example between 0.15% and 0.4%, e.g., between 0.2% and 0.3%.

[0048] The high water embodiments are preferably substantially free of organic solvents, having less than 5%, preferably less than 1%, more preferably no organic solvents. A preferred high-water embodiment was example Example 3, having a small amount of xylitol. Adding 45 parts ethylene glycol or 75 parts ethylene glycol to 100 parts of the composition of Example 3 did not reduce aluminum corrosion, but actually increased the corrosion of aluminum a small amount.

[0049] Adding acid to get to pH of below 1.5 significantly increased aluminum corrosion. Increasing the pH to with corrosion inhibitors like choline did not significantly protect the aluminum, but could ameliorate the affect of added phosphoric and/or phosphorous acid. An easier component to adjust pH was TMAH and TEA, and surprisingly, when the pH was adjusted to between 5 and 6 with either of these compounds the corrosion of aluminum was significantly reduced. Phosphonate-based corrosion inhibitors (e.g., nitrilotris(methylene phosphonic acid)) are effective at cutting metal corrosion in amounts below 0.5% even at very low (e.g., less than 1.5) pH values.

[0050] The compositions of Examples 3 to 18 can be used for single wafer cleaning applications, as they have been shown to exhibit a low TEOS etch rate and an acceptable Cu etch rate, cleaning times less than 1 minute, while being relatively environmentally friendly, relatively low cost, and relatively stable. In addition, these compositions may optionally include organic solvents, e.g., ethylene glycol, to augment resist-cleaning capability.

1 6. The method of any of claims 1 to 4, wherein the pH of the composition is from
2 about 3 to about 6.

1 7. The method of any of claims 1 to 6, further comprising the step of rinsing the
2 substrate.

1 8. The method of any of claims 6 to 7 wherein the composition comprises from
2 about 0.3% to about 4% of H_2SiF_6 .

1 9. The method of any of claims 6 to 7 wherein the composition comprises from
2 about 0.2% to about 2% of H_2SiF_6 .

1 10. A method of cleaning etching residue from a substrate comprising a low k
2 dielectric material comprising the steps of:

3 contacting the substrate with a composition comprising: (a) HBF_4 ; (b) an organic
4 solvent; (c) an amine; (d) a corrosion inhibitor; and (e) water, wherein said composition is
5 capable of exfoliating post-etch residues and of minimizing oxide loss, and wherein the
6 composition has a pH lower than 7, for a time and at a temperature sufficient to cause the
7 composition to substantially remove the residues.

1 11. The method of claim 10, wherein the composition comprises: (a) HBF_4 at a
2 concentration from about 0.001 % to about 5 % by weight of the composition; (b) the organic
3 solvent at a concentration from about 50 % to about 98 % by weight of the composition; (c)
4 the amine at a concentration less than about 1.5 % by weight of the composition; (d) the
5 corrosion inhibitor at a concentration from about 0.001 % to about 10 % by weight of the
6 composition; and (e) balance of water.

1 12. The method of claim 10, comprising: (a) HBF_4 at a concentration from about
2 0.05 % to about 3 % by weight of the composition; (b) the organic solvent at a concentration
3 from about 60 % to about 95 % by weight of the composition; (c) the amine at a
4 concentration less than about 1 % by weight of the composition; (d) the corrosion inhibitor at
5 a concentration from about 0.05 % to about 7 % by weight of the composition; and (e)
6 balance of water.

1 13. The method of claim 10, comprising: (a) HBF_4 at a concentration from about
2 0.1 % to about 2 % by weight of the composition; (b) the organic solvent at a concentration

1 24. The method of any of claims 17 to 23, wherein the composition is
2 substantially free of organic solvents.

1 25. The method of any of claims 17 to 23, wherein the composition consists
2 essentially of (a) H_2SiF_6 ; (b) water; and (c) a corrosion inhibitor.

1 26. The composition of any of claims 17 to 25, wherein the H_2SiF_6 is present in an
2 amount from about 0.2% to about 0.3% by weight.

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	WO 2004/030038 A (AIR PROD & CHEM ; EGBE MATTHEW (US)) 8 April 2004 (2004-04-08) page 1, line 5 - line 10 page 2, line 4 - line 13 page 4, line 11 - page 5, line 21; claims 1,2,12,13	10-16

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